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C08G 59/50**C08G 59/22****C08K 5/54****C08L 63/00**(21) Application number: **08324295**(71) Applicant: **SUMITOMO BAKELITE CO LTD**(22) Date of filing: **04.12.96**(72) Inventor: **OUNAMI KAZUTO**(54) **LIQUID EPOXY RESIN SEALING MATERIAL**

(57) Abstract:

PROBLEM TO BE SOLVED: To obtain a sealing material capable of remarkably improving the reliability of semiconductors in an acceleration test such as PCT (pressure-cooker test) and T/C (thermal cycle test) and useful for the sealing of a PGA-type semiconductor in contrast with conventional liquid sealing material giving insufficient reliability compared with a hermetic sealing with a ceramic material.

SOLUTION: This liquid epoxy resin sealing material is composed mainly of (A) a liquid epoxy resin, (B) a liquid alkylated diaminodiphenylmethane, (C) a polybutadiene having epoxy group and (D) an inorganic filler. The weight ratios of the components satisfy the following formulas; $(A)/[(A)+(B)]=0.65$ to 0.80 , $(C)/[(A)+(B)]=0.02$ to 0.05 and $(D)/[(A)+(B)+(C)+(D)]=0.50$ to 0.80 .

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No ratio
No solvent

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TITLE: Liquid epoxy resin potting material used especially for
potting semiconductors- comprising liquid epoxy resin,
liquid alkylated diaminodiphenylmethane, epoxidised
polybutadiene and inorganic filler

INVENTOR: KAZUTO, O; ONAMI, K

PATENT-ASSIGNEE: SUMITOMO BAKELITE KK[SUMB] , SUMITOMO BAKELITE CO LTD[SUMB]

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EP 846728 A2	June 10, 1998	E	007	C08L 063/00
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JP 10158365A	N/A	1996JP0324295	December 4, 1996
SG 60154A1	N/A	1997SG-0004092	November 19, 1997
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ABSTRACTED-PUB-NO: EP 846728A

BASIC-ABSTRACT:

A liquid epoxy resin potting material comprises: (a) a liquid epoxy resin; (b)

a liquid alkylated diaminodiphenylmethane; (c) an epoxidised polybutadiene; and (d) an inorganic filler. Weight ratios of individual components are: (a)/((a) + (b)) = 0.65-0.80 (I); (c)/((a) + (b)) = 0.01-0.05 (II); (d)/((a) + (b) + (c) + (d)) = 0.50-0.80.

Pref. at least 50%wt. (a) liquid epoxy resin has a viscosity of upto 8 Pa.s/25 deg. C. Inorganic filler (d) comprises a spherical silica having an average particle size of 0.1-3.0 μ m and a particle size of not more than 10 μ m; and a spherical silica of an average particle size of 410 μ m and a maximum size of upto 50 μ m. The compounding ratio is (a)/((a) + (b)) = 0.050.50.

USE - Material is used for potting ~~semiconductors~~ especially for PPGAType semiconductor that uses organic printed circuit board.

ADVANTAGE - Delamination does not occur when subjected to thermal stress. Moisture permeation and crack formation are reduced.

CHOSEN-DRAWING: Dwg.0/0

TITLE-TERMS: LIQUID EPOXY RESIN POTTING MATERIAL POTTING SEMICONDUCTOR COMPRISE
LIQUID EPOXY RESIN LIQUID ALKYLATED EPOXIDATION POLYBUTADIENE
INORGANIC FILL

DERWENT-CLASS: A21 A85 E16 E36 L03

CPI-CODES: A05-A01E2; A12-E04; A12-E07C; E10-B01A4; E31-P03; L04-C20A;

CHEMICAL-CODES:

Chemical Indexing M3 *01*

Fragmentation Code

G010 G019 G100 M1 M121 M132 M150 M280 M313 M321

M331 M340 M342 M414 M510 M520 M532 M540 M610 M782

M903 M904 Q454

Markush Compounds

199827-V0801-M

Chemical Indexing M3 *02*

Fragmentation Code

B114 B702 B720 B831 C108 C800 C802 C803 C804 C805

C807 M411 M782 M903 M904 M910 Q454 Q606

Specific Compounds

01694M

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1694U

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018 ; R00470 G1161 G1150 G1149 G1092 D01 D11 D10 D19 D18 D3 D50
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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the liquefied epoxy resin closure ingredient used for closure of a semi-conductor.

[0002]

[Description of the Prior Art] The flow of low-cost-izing in semi-conductor mounting and high integration has shifted to mounting gestalten, such as COB (chip onboard) and PPGA (plastics pin grid array), from DIP (dual inline package) by the conventional transfer molding. Although the liquefied closure ingredient is used for closure of a PPGA mold semi-conductor, compared with the hermetic seal mold by the ceramics, it is not enough in respect of dependability. As the cause, moisture invades from the organic printed wired board by which " package processing was carried out. In order unlike DIP by transfer molding to flow and to fabricate a liquefied closure ingredient by the non-draft, when air bubbles remain and heat stress is added, a crack occurs. Since the coefficient of linear expansion of a closure ingredient, and a semiconductor chip and an organic substrate differs, when heat stress is added, exfoliation will be produced in an interface and trespass of moisture will be made easy. " etc. was mentioned.

[0003]

[Problem(s) to be Solved by the Invention] In order to solve the conventional trouble, as a result of coming examination in piles wholeheartedly, this invention finds out that the constituent which blended the inorganic filler with a specific epoxy resin, aromatic series diamine, and an elastomer serves as a closure ingredient which can improve the dependability of a semi-conductor substantially in accelerated tests, such as PCT (pressure cooker test) and traveler's check (cold energy cycle test), and comes to be completed.

[0004]

[Means for Solving the Problem] In the liquefied epoxy resin closure ingredient with which this invention uses as a principal component (A) liquefied epoxy resin, (B) liquefied alkylation diamino diphenylmethane, the polybutadiene that has the (C) epoxy group, and (D) inorganic filler each -- a component -- the blending ratio of coal -- weight -- a ratio -- (-- A --) -- / -- [-- (-- A --) -- + -- (-- B --) --] -- = -- 0.65 - zero . -- 80 -- (-- C --) -- / -- [-- (-- A --) -- + -- (-- B --) --] -- = -- 0.02 - 0.05 -- And it is the liquefied epoxy resin closure ingredient characterized by being (D) / [(A)+(B)+(C)+(D)] = 0.50-0.80, and the dependability of the PPGA mold semi-conductor using an organic printed wired board can be improved substantially.

[0005]

[Embodiment of the Invention] In the liquefied epoxy resin used for this invention, it is desirable 50% of the weight or more of the component of that the viscosity in 25 degrees C is 8 or less Pa-s. Since air bubbles are involved in, or it will become easy to generate the short shot to a corner edge and will lead to dependability lowering in case the viscosity of a constituent becomes high and carries out inflow closure of the PPGA package with a liquefied closure ingredient if 50% of the weight or more of an

epoxy resin component is not epoxy of hypoviscosity, it is not desirable. As a viscosity measuring method of an epoxy resin, at a room temperature, when liquefied, in 25 degrees C, it measures with E mold viscometer [the product made from east machine industrial], and in a case solid at a room temperature, it measures at 150 degrees C using a high-temperature-service cone plate viscometer.

[0006] If it is the epoxy resin with which are satisfied of this requirement and an example will be given although not limited especially A bisphenol A diglycidyl ether mold epoxy resin, a bisphenol F diglycidyl ether mold epoxy resin, A bisphenol S diglycidyl ether mold epoxy resin, 3, 3', 5, 5'-tetramethyl - 4,4'-dihydroxybiphenyl-diglycidyl-ether mold epoxy resin, 1, 6-dihydroxy naphthalene diglycidyl ether mold epoxy resin, a phenol novolak mold epoxy resin, a cresol novolak mold epoxy resin, a bromination bisphenol A diglycidyl ether mold epoxy resin, a bromination cresol novolak mold epoxy resin, etc. -- it is -- these -- one sort -- or even if it mixes and uses, it does not interfere.

[0007] Unless it permutes 1 of the hydrogen of the benzene nucleus of diamino diphenylmethane, or two pieces or more by alkyl groups, such as a methyl group and an ethyl group, and the carbon number of an alkyl group is four or less, since it involves in air bubbles in case the viscosity of a constituent becomes high and the liquefied alkylation diamino diphenylmethane used for this invention carries out inflow closure of the package with a liquefied closure ingredient, or becomes easy to generate the short shot to a corner edge and leads to dependability lowering, it is not desirable. if it is the alkylation diamino diphenylmethane which satisfies this requirement and an example will be given, although it does not limit especially -- 3 and 3' -- the - diethyl -4 and - diamino diphenylmethane, 3, 3', and 4 '5, 5'-tetramethyl - 4 and 4 - diamino diphenylmethane, 3, 3', and '5, 5' - tetraethyl -4 and 4' - diamino diphenylmethane etc. -- it is -- these -- one sort -- or even if it mixes and uses, it do interfere.

[0008] liquefied -- an epoxy resin -- (-- A --) -- liquefied -- alkylation -- diamino -- diphenylmethane -- (-- B --) -- a weight mix -- a rate -- (-- A --) -- /-- [-- (-- A --) -- + -- (-- B --) --] -- = -- 0.65 - 0.80 -- the range -- it is -- things -- being desirable . (-- A --) -- /-- [-- (-- A --) -- + -- (-- B --) --] -- 0.80 -- exceeding -- if -- being unreacted -- an epoxy group -- increasing -- glass transition temperature -- etc. -- thermal resistance -- falling -- cold energy -- a cycle -- a trial -- dependability -- falling -- since -- not being desirable -- moreover -- 0.65 -- less than -- it is -- if -- a constituent -- shelf life -- falling -- since -- not being desirable .

[0009] 1000-5000 have [the polybutadiene (C) which has the epoxy group used for this invention] desirable number average molecular weight. Since viscosity will rise and workability will fall if the effectiveness of flexible grant becomes small and 5000 is exceeded, it is not desirable at less than 1000. Moreover, 3 - 10% of epoxy group content (principal chain addition mole fraction %) is desirable. At 3% or less, since it will become impossible to desire low stress-ization and the flexible grant effectiveness will become small in order to construct a bridge with a curing agent and not to take sea island structure if a chip and 10% are exceeded to compatibility with a liquefied epoxy resin (A), it is not desirable. an epoxy group -- having -- polybutadiene -- (-- C --) -- a weight mix -- a rate -- (-- C --) -- /-- [-- (-- A --) -- + -- (-- B --) --] -- = -- 0.01 - 0.05 -- the range -- carrying out -- things -- it is -- cold energy -- a cycle -- a trial -- the time -- generating -- heat stress -- a relaxation effect -- having . It is deficient in the improvement effectiveness of stress relaxation, and if 0.05 is exceeded, since [on the front face of a hardened material by lowering of the glass transition temperature of a hardened material, viscosity lifting of a constituent, and compatibility aggravation] it looms and ** occurs, it is not desirable at less than 0.01.

[0010] Fused silica, a crystal silica, etc. are mentioned as an inorganic bulking agent (D). Although a configuration generally has a globular shape, a letter of crushing, etc., in order to aim at reduction of coefficient of linear expansion and to add many bulking agents, a spherical bulking agent is the most desirable. As for an inorganic bulking agent (D), it is desirable that it is in the range of (D) / [(A)+(B)+ (C)+(D)] = 0.50-0.80 at a weight-mix rate. Since the viscosity of a constituent will rise and lowering of workability will be caused if the reduction effectiveness of coefficient of linear expansion is small and exceeds 0.80 less than by 0.50, it is not desirable. If a bulking agent with a small particle size is generally used, since it goes up and a fluidity falls, the contamination of air bubbles and a short shot will generate the viscosity of a resin constituent. On the other hand, if a bulking agent with a big particle size

is used, it will become easy to produce sedimentation and distribution of a bulking agent will become an ununiformity. If distribution of a bulking agent becomes an ununiformity, coefficient of linear expansion will differ in each part, or it will become easy to generate a crack in the upper part with few amounts of bulking agents. Moreover, if a maximum droplet size is larger than wire spacing, it will become easy to generate the short shot to the wire lower part, and short [by wire deformation / poor]. the above -- a problem -- solving -- a sake -- examination -- piling up -- particle size -- differing -- a silica -- namely, -- (-- a --) -- mean particle diameter -- 0.1 - 3.0 -- micrometer -- a maximum droplet size -- ten -- micrometer -- less than -- spherical -- a silica -- (-- b --) -- mean particle diameter -- four - ten -- micrometer -- a maximum droplet size -- 50 -- micrometer -- less than -- spherical -- a silica -- weight -- a ratio -- (-- a --) -- / -- (-- a --) -- + -- (-- b --) --] -- = -- 0.05 - 0.50 -- blending -- things -- a fluidity -- not spoiling -- sedimentation -- control -- being possible -- things -- having found out . not the thing that will be especially limited if it is the silica with which are satisfied of these requirements but (a), and (b) - one sort of each -- or even if it mixes and uses, it does not interfere. Particle size distribution and a mean diameter were measured by the laser formula (Horiba LA-500), and the mean diameter was taken as the median size. Even if it uses additives, such as the catalyst for promoting other resin and reactions other than said indispensable component if needed, a diluent, a pigment, a coupling agent, a leveling agent, and a defoaming agent, it does not interfere with the liquefied closure ingredient of this invention. A liquefied closure ingredient carries out distributed kneading of each component, the additive, etc. with 3 rolls, and manufactures by carrying out vacuum law evasion processing.

[0011]

[Example] This invention is concretely explained using an example below.

(Example 1) Distributed kneading of the following raw material was carried out with 3 rolls, vacuum degassing processing was carried out, and the liquefied epoxy resin closure ingredient was obtained. Using the obtained epoxy resin closure ingredient, the PPGA package was closed, it hardened in oven at 165 degrees C for 3 hours, and the semiconductor package was obtained. Moreover, the existence of an exfoliation and a crack with a semiconductor chip and a printed circuit board interface and sedimentation of a filler were checked by the following assessment approach, and the result was shown in a table 1.

- Bisphenol female mold epoxy resin: The 100 weight sections (161 or 25 degrees C [of weight per epoxy equivalent] viscosity 1.5 Pa-s)

- 3, the 3'-diethyl -4, the 4'-diamino diphenylmethane:40 weight section and epoxy denaturation polybutadiene : Six weight sections (number average molecular weight 1800, weight per epoxy equivalent 250)

- Spherical fused silica (1): The 340 weight sections (mean particle diameter of 6 micrometers, 48 micrometers of maximum droplet sizes)

- Spherical fused silica (2): 40 weight sections (mean particle diameter of 0.5 micrometers, 2 micrometers of maximum droplet sizes)

[0012] - Viscosity of a constituent : at restoration nature:80 degree C to E mold viscometer, 2.5rpm, and 25 degree C and a package, dispensing was carried out to the PPGA package and the restoration nature to a cavity was checked after 5 minutes.

- The existence check:(1) ordinary state of exfoliation and a crack (after hardening)

(2) The existence of an exfoliation and a crack with a semiconductor chip and a printed circuit board interface was checked with the supersonic detector (it is called Following SAT) about the 1000 cycle back for 125 degrees C of PCT (pressure cooker test) processings, -65 degrees C of 2.3 processings [atm and 168 hours after (3) traveler's-check (cold energy cycle test)], 30 minute <-->150 degrees C /, and 30 minutes.

- Filler sedimentation : the cross section of the package after hardening was carried out, the scanning electron microscope (it is called Following SEM) performed cross-section observation, and sedimentation from the hardened material upper part was measured. The things of an example 1 are not accepted to be also exfoliation and a crack, and its sedimentation of a filler is also very slight, and it became clear that it had good dependability. The number of packages used for each assessment is ten

pieces. The result is shown in a table 1.

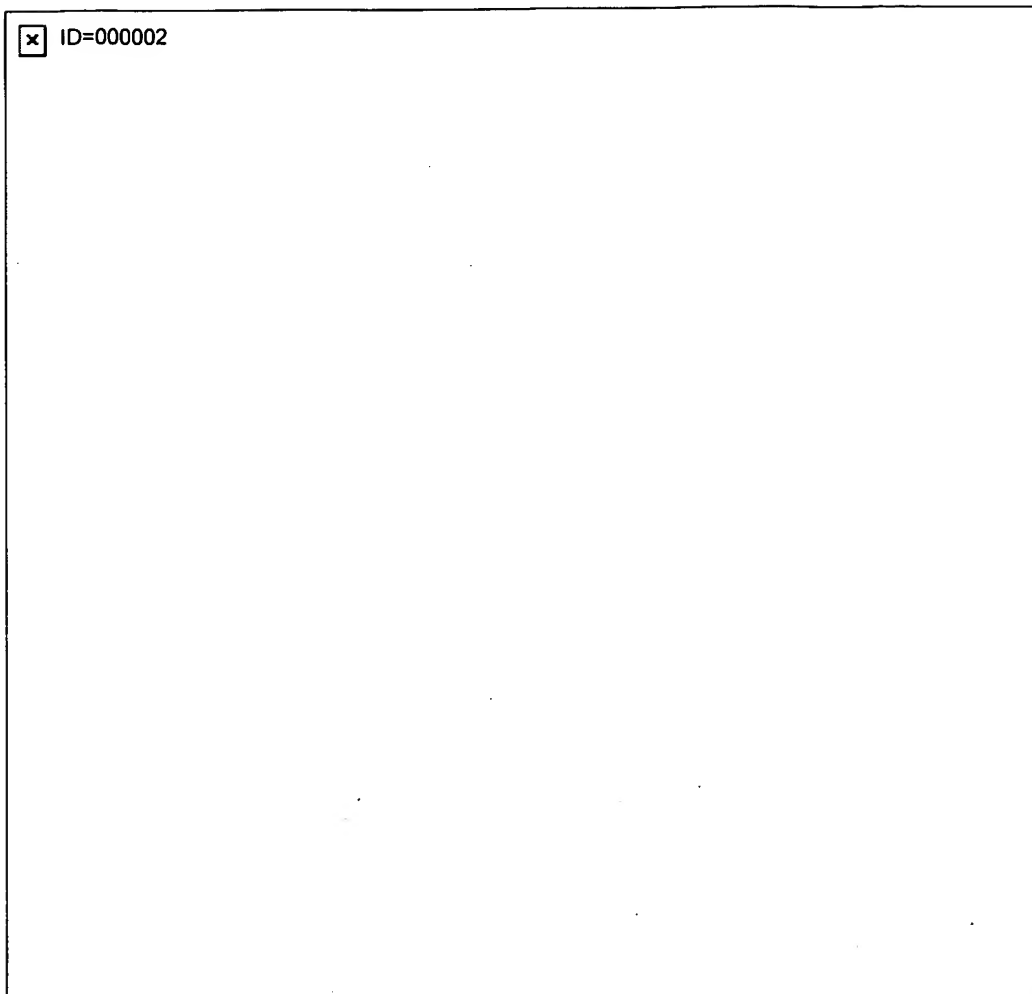
[0013] (Examples 2-8, examples 1-8 of a comparison) Except [all] this, the liquefied epoxy resin closure ingredient was obtained by the same approach as an example 1, the PPGA package was closed, and the combination formula shown in tables 1 and 2 estimated the dependability. The result is shown in tables 1 and 2.

[0014] The raw material used by the example 1 and others is the following object.

- Bisphenol female mold epoxy resin : (the Dainippon Ink & Chemicals, Inc. make, EXA-830LVP, 161 or 25 degrees C [of weight per epoxy equivalent] viscosity 1.5 Pa-s)
- The bisphenol A mold epoxy resin (1) : (the Dainippon Ink & Chemicals, Inc. make, EXA-850CRP, 171 or 25 degrees C [of weight per epoxy equivalent] viscosity 4.5 Pa-s)
- The bisphenol A mold epoxy resin (2) : (the product made from oil-ized Shell Epoxy, Epicoat 1001, weight per epoxy equivalent 470, solid)
- 1, 6-dihydroxy naphthalene mold epoxy resin : (the Dainippon Ink & Chemicals, Inc. make, HP-4032D, and 140 or 25 degrees C [of weight per epoxy equivalent] viscosity 25Paands)
- 3, the 3'-diethyl -4, 4'-diamino diphenylmethane : (the Nippon Kayaku Co., Ltd. make, kaya hard A-A)
- 3 3' - 5 5'-tetramethyl - 4 4'-diamino diphenylmethane : (the Nippon Kayaku Co., Ltd. make, kaya bond C-200)
- Epoxy denaturation polybutadiene : (the product made from Nippon Oil Chemistry, E-1800-6.5, number average molecular weight 1800, weight per epoxy equivalent 250)
- Spherical fused silica (1) : (the DENKI KAGAKU KOGYO K.K. make, FB-30, mean particle diameter of 6 micrometers, 48 micrometers of maximum droplet sizes)
- Spherical fused silica (2) : (Product made from ADOMA Tex, SO-25R, mean particle diameter of 0.5 micrometers, 2 micrometers of maximum droplet sizes)
- Spherical fused silica (3) : (Product made from ADOMA Tex, SO-32R, mean particle diameter of 2.0 micrometers, 8 micrometers of maximum droplet sizes)
- Spherical fused silica (4) : (the above-mentioned spherical fused silica (2), the mixture (weight ratio 1:1) of (3), mean particle diameter of 1.0 micrometers, 8 micrometers of maximum droplet sizes)
- Spherical fused silica (5) : (the DENKI KAGAKU KOGYO K.K. make, FB-25S, mean particle diameter of 17 micrometers, 75 micrometers of maximum droplet sizes)

[0015]

[A table 1]



[0016]
[A table 2]

表2

	比較例					
	1	2	3	4	5	6
配合(重量部)						
ビスフェノールF型エポキシ樹脂	100	100	100	100	100	30
ビスフェノールA型エポキシ樹脂(2)						70
3,3'-ジエチル-4,4'-ジアミノジフェニルメタン	40	40	40	40	40	40
エポキシ変性ポリブタジエン	6	6		6	6	6
球状溶融シリカ(1)	380	420	230	110		340
球状溶融シリカ(2)		280	160	260		40
球状溶融シリカ(5)					380	
特性						
組成物粘度(Pa・s)	30	120	17	130	27	125
パッケージ充填性	良好	不良	良好	不良	良好	不良
剥離						
硬化後	0	—	0	—	0	—
PCT後	0	—	8	—	0	—
T/C後	3	—	0	—	4	—
クラック						
硬化後	0	—	0	—	0	—
PCT後	0	—	3	—	0	—
T/C後	5	—	0	—	8	—
フィルター沈降(μm)	80	—	3以下	—	100	—

[0017]

[Effect of the Invention] If a semiconductor package is closed with the liquefied closure ingredient of this invention, since the semiconductor package of high-reliability without exfoliation and a crack can be obtained in a pressure cooker test or a cold energy cycle test, without spoiling a fluidity and workability, a industrial merit is large.

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CLAIMS

[Claim(s)]

[Claim 1] (A) In liquefied epoxy resin and (B) liquefied alkylation diamino diphenylmethane, the polybutadiene which has the (C) epoxy group, and the liquefied epoxy resin closure ingredient which uses (D) inorganic filler as a principal component each -- a component -- the blending ratio of coal -- weight -- a ratio -- (-- A --) -- /-- [-- (-- A --) -- + -- (-- B --) --] -- = -- 0.65 - zero . -- 80 -- (-- C --) -- /-- [-- (-- A --) -- + -- (-- B --) --] -- = -- 0.01 - 0.05 -- it is -- and -- (-- D --) -- /-- [-- (-- A --) -- + -- (-- B --) -- + -- (-- C --) -- + -- (-- D --) --] -- = -- 0.50 - 0.80 -- it is -- things -- the description -- ** -- carrying out -- liquefied -- an epoxy resin -- closure -- an ingredient .

[Claim 2] (A) The liquefied epoxy resin closure ingredient according to claim 1 whose viscosity of at least 50% of the weight of the component of a liquefied epoxy resin is 8 Pa-s / 25 degrees C or less.

[Claim 3] (-- D --) -- inorganic -- a filler -- (-- a --) -- mean particle diameter -- 0.1 - 3.0 -- micrometer -- a maximum droplet size -- ten -- micrometer -- less than -- spherical -- a silica -- (-- b --) -- mean particle diameter -- four - ten -- micrometer -- a maximum droplet size -- 50 -- micrometer -- less than -- spherical -- a silica -- from -- becoming -- the blending ratio of coal -- weight -- a ratio -- (-- a --) -- /-- [- (-- a --) -- + -- (-- b --) --] -- = -- 0.05 - 0.50 -- it is -- being according to claim 1 -- liquefied -- an epoxy resin -- closure -- an ingredient .

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